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## (54) EXHAUST GAS PURIFICATION FILTER, EXHAUST GAS PURIFICATION CATALYST, AND ITS MANUFACTURING METHOD

(57)Abstract:

PROBLEM TO BE SOLVED: To satisfy both of a low pressure loss and a high PM capturing ratio in a diesel engine.

SOLUTION: The surface pores to be opened to the surface of a filter bulkhead are formed so that the total opening area of surface pores having pore diameters of 40 $\mu$ m or less is 50% or more of the total opening area of all the surface pores. The distribution of surface pores in the filter bulkhead can be optimized to enhance the capturing ratio of PM while suppressing the rise of exhaust pressure loss.

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**CLAIMS**

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[Claim(s)]

[Claim 1]

It is the emission-gas-purification filter which serves as more a filter septum which divides the inflow side cel by which the weather strip was carried out by the exhaust gas downstream, the outflow side cel by which adjoined this inflow side cel and the weather strip was carried out by the exhaust gas upstream, and this inflow side cel and this outflow side cel,

The emission-gas-purification filter with which an aperture is characterized by occupying 50% or more of the sum total opening area of all surface holes by the sum total opening area of a surface hole 40 micrometers or less among the surface holes as for which the pore of this filter septum carried out opening to the front face.

[Claim 2]

The emission-gas-purification filter according to claim 1 with which the internal pore whose aperture is 20-40 micrometers among the internal pores of said filter septum occupies 50% or more of all internal pores.

[Claim 3]

The catalyst for emission gas purification characterized by having the catalyst bed which supported the catalyst metal in said filter septum of an emission-gas-purification filter according to claim 1 or 2.

[Claim 4]

The filter septum which divides the inflow side cel by which the weather strip was carried out by the exhaust gas downstream, the outflow side cel by which adjoined this inflow side cel and the weather strip was carried out by the exhaust gas upstream, and this inflow side cel and this outflow side cel, and the catalyst bed formed in this filter septum are included,

The catalyst for emission gas purification characterized by for an aperture inclining toward larger large pore than a predetermined value among the internal pores of this filter septum, and forming this catalyst bed.

[Claim 5]

Like the packer whom an aperture inclines toward the small pore below a predetermined value, is filled up with combustible material, and uses as a restoration emission-gas-purification filter among the internal pores of the filter septum which divides the inflow side cel by which the weather strip was carried out by the exhaust-gas downstream, the outflow side cel by which adjoined this inflow side cel and the weather strip was carried out by the exhaust-gas upstream, and this inflow side cel and this outflow side cel, and this filter septum of the emission-gas-purification filter containing two or more cels which become more,

The catalyst bed formation process which forms the catalyst bed which contains a porosity oxide and noble metals in this filter septum of this restoration emission-gas-purification filter at least,

The manufacture approach of the catalyst for emission gas purification characterized by including the destruction-by-fire process which makes this combustible material with which the pore of this filter septum is filled up burned down.

[Claim 6]

The manufacture approach of the catalyst for emission gas purification according to claim 5 of performing the removal process which removes physically said combustible material with which larger large pore than said predetermined value was filled up with the aperture like said packer.

[Claim 7]

Said combustible material is the manufacture approach of the catalyst for emission gas purification according to claim 5 that mean particle diameter is the inflammable powder below said predetermined value.

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**DETAILED DESCRIPTION**

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[Detailed Description of the Invention]

[0001]

[Field of the Invention]

This invention relates to the emission-gas-purification filter which purifies the exhaust gas containing a particulate, such as exhaust gas from a diesel power plant, and the catalyst for emission gas purification and its manufacture approach.

[0002]

[Description of the Prior Art]

About the gasoline engine, the injurious ingredient in exhaust gas has been decreasing certainly by severe regulation of exhaust gas, and advance of the technique in which it can be coped with. However, about the diesel power plant, the advance of a technique is also behind also in regulation from the unique situation that an injurious ingredient is discharged as a particulate (particulate matter: sulfur system particles, such as a carbon particle and sulfate, the amount hydrocarbon particle of macromolecules, etc. are called following PM), compared with the gasoline engine.

[0003]

As an exhaust gas purge for diesel power plants currently developed by current, it roughly divides and the emission-gas-purification filter (Wall flow) of a trap mold and the exhaust gas purge (straight flow) of an open type are known. Among these, as an emission-gas-purification filter of a trap mold, the \*\*\*\*\* type honeycomb object made from a ceramic (diesel PM filter (it is called Following DPF)) is known. Discharge controls by this DPF consisting of a filter septum which divides the inflow side cel by which comes to \*\*\*\*\* the both ends of opening of the cel of a ceramic emission-gas-purification filter in checkers by turns, and the weather strip was carried out by the exhaust-gas downstream, the outflow side cel by which adjoined the inflow side cel and the weather strip was carried out by the exhaust-gas upstream, and an inflow side cel and an outflow side cel, filtering exhaust gas by the pore of a filter septum, and carrying out uptake of the PM.

[0004]

However, since a pressure loss goes up by deposition of PM, it is necessary to remove periodically PM deposited with a certain means, and to reproduce in DPF. Then, when a pressure loss goes up conventionally, reproducing DPF by burning PM deposited in the burner or the electric heater is performed. However, in this case, the temperature at the time of combustion rises, so that there is much alimentation of PM, and DPF may be damaged with the thermal stress by it.

[0005]

So, in recent years, a coat layer is formed in the front face of the filter septum of DPF from an alumina etc., and the continuation playback type DPF which has the catalyst bed which supported catalyst metals, such as platinum (Pt), in the coat layer is developed. Since PM by which uptake was carried out carries out oxidation combustion by the catalytic reaction of a catalyst metal according to this continuation playback type DPF, DPF is reproducible by making it burn simultaneous in uptake succeeding uptake. And since catalytic reaction can burn while there are few being comparatively generated at low temperature and amounts of uptake, it has the advantage that the thermal stress which acts on DPF is small, and breakage is prevented.

[0006]

As such a continuation playback type DPF, the thing of a configuration of that, as for the porosity oxide with which an average aperture is 5-35 micrometers, and the porosity of a filter septum constitutes a catalyst bed from 40 - 65%, the thing of a particle size smaller than the average aperture of a filter septum occupies

90 % of the weight or more is indicated by JP,09-220423,A. A catalyst bed can be formed even in the internal surface of not only the front face of a filter septum but pore by carrying out the coat of such a porosity oxide of high specific surface area. Moreover, since thickness of fixed, then a catalyst bed can be made thin for the amount of coats, increase of a pressure loss can be controlled.

[0007]

Moreover, in JP,06-159037,A, it is NO<sub>x</sub> further to the above-mentioned catalyst bed. The continuation playback type DPF which supported occlusion material is indicated. It will be NO<sub>x</sub> if it does in this way. It is NO<sub>x</sub> to occlusion material. NO<sub>x</sub> by which occlusion was carried out by being able to carry out occlusion and spraying reducing agents, such as gas oil, It becomes possible to return and purify.

[0008]

[Patent reference 1] JP,09-220423,A

[Patent reference 2] JP,06-159037,A

[0009]

[Problem(s) to be Solved by the Invention]

However, if the amount of PM which will be deposited in conventional DPF if PM is contained so much in exhaust gas increases rapidly, an exhaust air pressure loss goes up, it is going to control this and the porosity of a filter septum is raised, PM which passes through a filter septum will increase and the collection efficiency of PM will fall. That is, since an exhaust air pressure loss and the collection efficiency of PM had the relation of a rebellion event, they were difficult to satisfy both only to control of porosity.

[0010]

Moreover, in the continuation playback type DPF which formed the catalyst bed also in the internal surface of the pore of a filter septum, the pore of a filter septum blockaded by the catalyst bed, consequently faults, such as increase of an exhaust air pressure loss and a fall of PM oxidation ability, have arisen. And if the amount of coats of a catalyst bed is controlled to extent by which pore is not blockaded, while the purification engine performance will fall, since a support consistency becomes high, there is a problem that platinum etc. carries out grain growth and activity falls at the time of elevated-temperature durability.

[0011]

While this invention is made in view of such a situation and making an exhaust air pressure loss small, it sets it as one purpose to offer DPF with the high collection efficiency of PM. Moreover, another purpose of this invention is to control increase of a pressure loss while forming sufficient quantity of a catalyst bed.

[0012]

[Means for Solving the Problem]

The description of the emission-gas-purification filter of this invention which solves the above-mentioned technical problem The inflow side cel by which the weather strip was carried out by the exhaust gas downstream, and the outflow side cel by which adjoined the inflow side cel and the weather strip was carried out by the exhaust gas upstream, It is the emission-gas-purification filter which serves as more a filter septum which divides an inflow side cel and an outflow side cel, and an aperture is among the surface holes as for which the pore of a filter septum carried out opening to the front face for the sum total opening area of a surface hole 40 micrometers or less to occupy 50% or more of the sum total opening area of all surface holes.

[0013]

In the above-mentioned emission-gas-purification filter, it is desirable for the internal pore whose aperture is 20-40 micrometers among the internal pores of a filter septum to occupy 50% or more of all internal pores.

[0014]

And the description of one catalyst for emission gas purification of this invention is to have the catalyst bed which supported the catalyst metal in the filter septum of the emission-gas-purification filter of above-mentioned this invention.

[0015]

Moreover, the description of another catalyst for emission gas purification of this invention which solves the above-mentioned technical problem The inflow side cel by which the weather strip was carried out by the exhaust gas downstream, and the outflow side cel by which adjoined the inflow side cel and the weather strip was carried out by the exhaust gas upstream, It is in an aperture inclining toward larger large pore than a predetermined value among the pores of a filter septum, and the catalyst bed being formed including the filter septum which divides an inflow side cel and an outflow side cel, and the catalyst bed formed in the filter septum.

[0016]

And the description of the manufacture approach of this invention that the above-mentioned catalyst for emission gas purification can be manufactured The inflow side cel by which the weather strip was carried out by the exhaust gas downstream, and the outflow side cel by which adjoined the inflow side cel and the weather strip was carried out by the exhaust gas upstream, Like the packer whom an aperture inclines toward the small pore below a predetermined value, is filled up with combustible material, and uses as a restoration emission-gas-purification filter among the pores of the filter septum which divides an inflow side cel and an outflow side cel, and the filter septum of the emission-gas-purification filter containing two or more cels which become more It is in including the catalyst bed formation process which forms the catalyst bed which contains a porosity oxide and noble metals in the filter septum of a restoration emission-gas-purification filter at least, and the destruction-by-fire process which makes the combustible material with which the pore of a filter septum is filled up burned down.

[0017]

In the above-mentioned manufacture approach, it is desirable to perform the removal process which removes physically the combustible material with which larger large pore than said predetermined value was filled up with the aperture like the packer.

[0018]

[Embodiment of the Invention]

The surface hole the pore of a filter septum carried out [ the hole ] opening to the front face exists in a filter septum, and the surface hole is open for free passage to the internal pore of a filter septum. And uptake of the bigger PM than the aperture of a surface hole is carried out to the front face of a filter septum, PM smaller than the aperture of a surface hole advances into internal pore from a surface hole, and uptake is carried out to internal pore. Here, if the aperture of a surface hole is too large, since the amount of PM by which uptake is carried out to the front face of a filter septum decreases, the collection efficiency of PM will become low. Moreover, if the aperture of a surface hole is too small, since a surface hole will be closed by PM by which uptake was carried out to the front face of a filter septum, an exhaust air pressure loss goes up.

[0019]

So, with the emission-gas-purification filter of this invention, the sum total opening area of a surface hole 40 micrometers or less forms [ the aperture ] 50% or more of the sum total opening area of all surface holes among the surface holes as for which the pore of a filter septum carried out opening to the front face. It becomes the best for the front face of a filter septum distributing [ which carries out opening / of a surface hole ] this, and it becomes possible to raise the collection efficiency of PM, controlling the rise of an exhaust air pressure loss. Fault comes to arise that an aperture is [ surface holes 40 micrometers or less ] less than 50% of all surface holes in the collection efficiency of PM, either of the exhaust air pressure losses, or both.

[0020]

The thing of the sum total opening area of the surface hole to the total surface area of a filter septum considered comparatively (surface numerical aperture) as 25 - 35% of range is desirable. If a surface numerical aperture is smaller than 25%, an exhaust air pressure loss will go up, if a surface numerical aperture becomes large from 35%, there will be many amounts of PM which pass through a filter septum, and the collection efficiency of PM will fall.

[0021]

In the emission-gas-purification filter of this invention, it is desirable for the internal pore whose aperture is 20-40 micrometers among the internal pores of a filter septum to occupy 50% or more of all internal pores. If the large pore to which an aperture exceeds [ the internal pore whose aperture is 20-40 micrometers ] 40 micrometers by less than 50% of all internal pores increases, there will be many amounts of PM which pass through a filter septum, the collection efficiency of PM will fall, and if the aperture of small pore [ less than 20-micrometer ] increases, a pressure loss will increase.

[0022]

Moreover, the thing of the sum total volume of the internal pore to the volume of a filter septum considered comparatively (porosity) as 60 - 65% of range is desirable. If porosity is smaller than 60%, an exhaust air pressure loss will go up, if porosity becomes large from 65%, there will be many amounts of PM which pass through a filter septum, and the collection efficiency of PM will fall.

[0023]

Although the aperture and rate of the internal pore of a filter septum can be measured by the press fit measuring method by the mercury porosimeter etc., it is [ the aperture and rate of a surface hole ] desirable to ask by the direct observational method under a microscope etc. By the press fit measuring method, the

measurement about a surface hole is difficult.

[0024]

With one catalyst for emission gas purification of this invention, the catalyst bed which supported the catalyst metal to the filter septum of the emission-gas-purification filter of this invention is formed. Since there are few osculum to which a surface hole 40 micrometers or less has many apertures, and an aperture exceeds 40 micrometers in the front face of a filter septum, PM is first distributed on the whole front face of a filter septum. And an aperture advances into internal pore from a surface hole 40 micrometers or less, contacts a catalyst bed, and burns with a catalyst metal. Deposition of PM is controlled by this and the rise of an exhaust air pressure loss is also prevented.

[0025]

And if the internal pore whose aperture is 20-40 micrometers forms 50% or more of all internal pores, since the collection efficiency of PM will increase, while it passes through and lock out of pore is controlled, sequential combustion removal of the PM is carried out.

[0026]

In order to form a catalyst bed in a filter septum, the wash coat of the slurry of support powder, such as an alumina, is carried out, and after calcinating it, the approach of supporting Pt etc. is adopted. And a catalyst bed can be formed even in the internal surface of not only the front face of a filter septum but internal pore by carrying out the coat of the support powder with which the thing of a particle size smaller than the average aperture of a filter septum occupies 90 % of the weight or more as indicated by JP,9-220423,A. Moreover, since thickness of fixed, then a catalyst bed can be made thin for the amount of coats, increase of a pressure loss can be controlled.

[0027]

However, since support powder has a fine particle size, it invades in the interior pore of size \*\*\*\*\* of an aperture, and it is got blocked one by one from the small small pore of an aperture. If it remains as it is, the opening area of the surface hole which carries out opening to a filter septum will become small, and a pressure loss will go up. Then, after carrying out the coat of the slurry, making an excessive slurry discharge from internal pore by suction or the air blow is performed. Even if it is difficult to remove a slurry from small pore, while a slurry is discharged from the big large pore of an aperture and a coat layer is formed in the internal surface of internal pore by doing in this way, since the interior serves as a cavity and can also secure the opening area of a surface hole, it can make a pressure loss low.

[0028]

Purification of PM by the continuation playback type DPF is performed by the principle by which oxidation purification is carried out because PM by which uptake was carried out into internal pore contacts Pt of a catalyst bed etc. However, as described above, when the great portion of internal pore is the big large pore of an aperture, collision frequency with the catalyst bed currently formed in the internal surface is low [ PM ] in order to pass large pore. Therefore, PM which passes through a filter septum will increase and the purification engine performance of PM will become low.

[0029]

On the other hand, the amount of coats is lessened, lock out of small pore is controlled, the uptake place of PM, then the collection efficiency of PM improve small pore, and the purification engine performance of PM improves. However, since the thickness of a catalyst bed becomes thin, if the amounts of support, such as platinum, are made equivalent, a support consistency will become high too much, and there is fault that carry out grain growth and activity falls at the time of elevated-temperature durability.

[0030]

So, with another catalyst for emission gas purification of this invention, among the internal pores of a filter septum, an aperture inclines toward larger large pore than a predetermined value, and forms the catalyst bed. Since what is contributed to especially the uptake of PM is internal pore whose aperture is 20-40 micrometers, as a predetermined value, the value of 40 micrometers or less is desirable, and especially the thing for which the value of 20 micrometers or less is adopted is desirable. For example, since 20 micrometers, then an aperture are not formed in a predetermined value at small pore 20 micrometers or less and the catalyst bed is not blockaded, small pore serves as a path of exhaust gas, and can make initial pressure loss low. Moreover, even if PM accumulates on small pore, an aperture can maintain a pressure loss low by bigger large pore than 20 micrometers. And since the catalyst bed is formed in bigger large pore than 20 micrometers, PM by which uptake was carried out to the large pore contacts a catalyst bed, and combustion purification is efficiently carried out.

[0031]

In order to manufacture the catalyst for emission gas purification of this invention, the emission-gas-purification filter containing two or more cells which serve as more a filter septum which divides the inflow side cell by which the weather strip was first carried out by the exhaust gas downstream, the outflow side cell by which adjoined the inflow side cell and the weather strip was carried out by the exhaust gas upstream, and an inflow side cell and an outflow side cell is prepared. Conventional DPF may be used as it is and the emission-gas-purification filter containing the straight cell by which a weather strip is not carried out at both ends depending on the case may be used.

[0032]

Next, among the internal pores of the filter septum of an emission-gas-purification filter, an aperture inclines toward the small pore below a predetermined value, and is filled up with combustible material. As combustible material, the liquefied organic substance, such as an organic solution which dissolved solid-state organic substance powder [ , such as carbon powder, cellulose powder, starch, and resin powder, ] or surfactant, liquefied resin, and acrylic oligomer, an organic plasticizer, and the organic substance in the organic solvent, can be used. When using solid-state organic substance powder as combustible material, and mean particle diameter uses the powder below said predetermined value, an aperture can incline and fill up the small pore below a predetermined value.

[0033]

Moreover, since a restoration condition is maintained with surface tension in pore with a small aperture although almost all interior pore is filled up with the liquefied organic substance when using the liquefied organic substance as combustible material, and it is easy to flow out of pore with a big aperture, an aperture inclines toward the small pore below a predetermined value, and it can be filled up.

[0034]

In addition, it is as desirable as a packer to perform the removal process which removes physically the combustible material with which it filled up. For example, by performing suction or an air blow, an aperture can remove more certainly the combustible material with which larger large pore than a predetermined value is filled up. Moreover, a restoration emission-gas-purification filter is vibrated, an outflow can be promoted, or it can be made to be able to rotate and a centrifugal force can also remove.

[0035]

In a catalyst bed formation process, a catalyst bed is continuously formed [ an aperture ] by the restoration emission-gas-purification filter with which combustible material inclined at the small pore below a predetermined value, and it filled up with it. This catalyst bed comes to support a catalyst metal to a porosity oxide, and is a porosity oxide. The multiple oxide which consists of oxides, such as aluminum  $2O_3$ , and  $ZrO_2$ ,  $CeO_2$ ,  $TiO_2$ ,  $SiO_2$ , or two or more of these sorts can be used.

[0036]

This catalyst bed is formed not only in the front face of a filter septum but in the internal surface of the large pore to which the aperture with which combustible material is not filled up exceeds said predetermined value.

[0037]

It is desirable to consider as the catalyst bed currently formed in this filter septum and the amount of coats of 100 - 200 g/L. the amount of coats in less than 100 g/L, the fall of the endurance by grain growth of a catalyst metal is not avoided -- a pressure loss becomes high too much and is not practical if 200 g/L is exceeded.

[0038]

What is necessary is to make oxide powder or multiple oxide powder into a slurry with a binder component and water, such as alumina sol, and just to calcinate, after making the slurry adhere to a filter septum in order to form a catalyst bed. Although dip coating usual to making a slurry adhere to a filter septum can be used, it is desirable to remove the extraneous article of a slurry which entered in pore by the air blow or suction.

[0039]

Although it can use as a catalyst metal supported by the catalyst bed if oxidation of PM is promoted by catalytic reaction, it is desirable to support a kind chosen from the noble metals of platinum groups, such as Pt, Rh, and Pd, at least or two or more sorts. Furthermore, it is  $NO_x$ . It is also desirable to support occlusion material. As for the amount of support of noble metals, it is desirable to consider as the range of per [ 2-8g ] volume of 1l. of an emission-gas-purification filter. It will become a cost rise while activity is saturated, even if activity will be too low, will not be practical and will support mostly from this range, if there are few amounts of support than this.

[0040]

Moreover, what is necessary is just to support using the solution which dissolved the nitrate of noble metals etc. in the coat layer which consists of oxide powder or multiple oxide powder by the adsorption supporting method, the water absorption supporting method, etc., in order to support noble metals. Moreover, noble metals are beforehand supported to oxide powder or multiple oxide powder, and a catalyst bed can also be formed using the catalyst powder.

[0041]

NO<sub>x</sub> which can be supported to a catalyst bed As occlusion material, it can choose from rare earth elements, such as alkaline earth metal, such as alkali metal, such as K, Na, Cs, and Li, and Ba, calcium, Mg, Sr, or Sc, Y, Pr, Nd, and can use. It is NO<sub>x</sub> especially. The thing of the alkali metal which excelled in occlusion ability, and alkaline earth metal for which a kind is used at least is desirable.

[0042]

This NO<sub>x</sub> As for the amount of support of occlusion material, it is desirable to consider as the range of 0.25-0.45 mols per volume of 1l. of an emission-gas-purification filter. If activity is too low and it is not practical, if there are few amounts of support than this, and it supports mostly from this range, noble metals will be covered and activity will come to fall.

[0043]

Moreover, NO<sub>x</sub> What is necessary is just to support in a coat layer by the water absorption supporting method etc. using the solution which dissolved acetate, a nitrate, etc., in order to support occlusion material. Moreover, it is NO<sub>x</sub> beforehand to oxide powder or multiple oxide powder. Occlusion material is supported and a catalyst bed can also be formed using the powder.

[0044]

Next, the destruction-by-fire process which makes the combustible material with which the internal pore of a filter septum is filled up burned down is performed. Since the small pore below said predetermined value serves as a cavity, and the aperture in which the catalyst bed is not formed carries out opening and functions as an exhaust gas path by combustible material being burned down at the time of use, increase of a pressure loss can be controlled. This destruction-by-fire process may be performed to baking and coincidence of a slurry by which the coat was carried out, and may perform baking of a slurry independently.

[0045]

In addition, if what contains a metal ion as combustible material is used, a metal may remain to a filter septum also after destruction by fire, and it may act as a catalyst.

[0046]

[Example]

Hereafter, the example of a trial, an example, and the example of a comparison explain this invention concretely.

[0047]

(Example of a trial)

Two or more kinds of DPF base materials made from cordierite (2L) with which pore distribution of a filter septum differs were prepared, and surface hole distribution of the filter septum and internal pore distribution were measured, respectively. Surface hole distribution was measured by the image analysis of a microphotography, and internal pore distribution was measured by the mercury porosimeter.

[0048]

Next, the wash coat of the slurry which is mainly concerned with alumina powder with a mean particle diameter of 1-3 micrometers was carried out to each DPF base material, and suction removal of the excessive slurry was carried out. It ranks second. It calcinated at 450 degrees C after desiccation by 110 degrees C, and the coat layer was formed. A coat layer is per 1l. of each DPF base material. 150g was formed. the specified quantity of the dinitrodiammine platinum solution of predetermined concentration is infiltrated into a coat layer after that -- 120 degree C -- after 1-hour desiccation It calcinated at 500 degrees C for 1 hour, Pt was supported in the coat layer, and the catalyst bed was formed. The amount of support of Pt is 2g per 1l. of each DPF base material.

[0049]

Two or more acquired catalysts for emission gas purification were packed in the case, respectively, and were catalytic-converter-ized. These were attached in the exhaust air system of 2L direct injection diesel power plant on the engine bench, respectively, and the exhaust air pressure loss and PM collection efficiency of 3 hours after when operating by 2000rpm x30Nm were measured, respectively. In addition, PM collection efficiency came out with the amount of PM in entering gas, and was computed from the ratio of the amount

of PM in gas. And measured value is arranged according to surface hole distribution or internal pore distribution, and a result is shown in drawing 1 - drawing 10 .

[0050]

first -- a surface hole -- comparatively -- \*\* -- if it sees about relation with an exhaust air pressure loss -- the aperture among the surface holes of drawing 1 and drawing 2 to a filter septum -- a surface hole 40 micrometers or less -- comparatively -- \*\* -- if a pressure loss becomes low and an aperture is [ a surface hole 40 micrometers or less ] 50% or more so that a negative correlation is between pressure losses and the rate of a surface hole 40 micrometers or less becomes [ an aperture ] high -- pressure loss It is thought that it is set to 15 or less kPas. On the other hand, a correlation is not accepted between the rate of the surface hole where an aperture exceeds 40 micrometers, and a pressure loss.

[0051]

moreover, a surface hole -- comparatively -- \*\* -- if it sees about relation with PM collection efficiency -- the aperture among the surface holes of drawing 3 and drawing 4 to a filter septum -- a surface hole 40 micrometers or less -- comparatively -- \*\* -- if PM collection efficiency becomes high and an aperture is [ a surface hole 40 micrometers or less ] 50% or more so that a forward correlation is between PM collection efficiency and the rate of a surface hole 40 micrometers or less becomes [ an aperture ] high -- PM collection efficiency -- almost -- Becoming 100% is admitted. On the other hand, a correlation is not accepted between the rate of the surface hole where an aperture exceeds 40 micrometers, and a pressure loss.

[0052]

therefore -- if the sum total opening area of a surface hole 40 micrometers or less occupies [ an aperture ] 50% or more of the sum total opening area of all surface holes -- pressure loss 15 or less kPas -- low -- PM collection efficiency -- almost -- It is clear to become 100%.

[0053]

next, internal pore -- comparatively -- \*\* -- the internal pore whose aperture is 20-40 micrometers among the internal pores of a filter septum from drawing 5 - drawing 7 when it sees about relation with an exhaust air pressure loss -- comparatively -- \*\* -- if the percentage of internal pore that a negative correlation is between pressure losses and an aperture is 20-40 micrometers is 50% or more -- a pressure loss -- number It can be made very small with kPa. However, a correlation forward in an aperture is accepted between the rate of less than 20-micrometer internal pore, and a pressure loss, and a pressure loss will increase, so that an aperture makes [ many ] less than 20-micrometer internal pore. On the other hand, a correlation is not accepted between the rate of internal pore that an aperture exceeds 40 micrometers, and a pressure loss.

[0054]

moreover, internal pore -- comparatively -- \*\* -- the internal pore whose aperture is 20-40 micrometers among the internal pores of a filter septum from drawing 8 - drawing 10 when it sees about relation with PM collection efficiency -- comparatively -- \*\* -- if the percentage of internal pore that a forward correlation is between PM collection efficiency, and an aperture is 20-40 micrometers is 50% or more -- PM collection efficiency -- almost -- It is 100%. However, a negative correlation is accepted between the rate of internal pore that an aperture exceeds less than 20 micrometers or 40 micrometers, and PM collection efficiency, and PM collection efficiency will fall, so that internal pore to which an aperture exceeds less than 20 micrometers or 40 micrometers is made [ many ] .

[0055]

therefore -- if the internal pore whose aperture is 20-40 micrometers occupies 50% or more of all internal pores -- a pressure loss -- number below kPa -- low -- PM collection efficiency -- almost -- It is 100%.

[0056]

In addition, from the data of drawing 1 - drawing 10 , the degree (contribution) to which surface hole distribution or distribution of internal pore affects a pressure loss or PM collection efficiency is calculated, and a result is shown in drawing 11 . In addition, the count approach followed the multiple regression analysis of the multivariate-analysis technique.

[0057]

The effect the direction of distribution of a surface hole affects a pressure loss and PM collection efficiency is larger than drawing 11 , and it turns out that distribution of a surface hole has contributed to especially PM collection efficiency greatly.

[0058]

(Example 1)

The 2l. DPF base material made from cordierite was prepared, and it has arranged in the atmospheric-air

ambient atmosphere in which the carbon powder of 10 micrometers of mean diameters is flowing by the flow rate of 1 g/hr, and processed for 1 hour. Since the air containing carbon flows into an inflow side cel, a filter septum is passed and it flows out of an outflow side cel, uptake of the carbon powder is carried out into the pore of a filter septum between them.

[0059]

Since the mean particle diameter of the carbon powder 2 is 10 micrometers as shown in drawing 12, it is easy to pass through the pore 10 to which the aperture of the filter septum 1 exceeds 10 micrometers, and an aperture deposits it on the pore 11 10 micrometers or less gradually, and it is filled up with the pore 11.

[0060]

Next, the wash coat of the slurry which is mainly concerned with alumina powder with a mean particle diameter of 1-3 micrometers was carried out, and suction removal of the excessive slurry was carried out. As shown in drawing 13, the aperture of the filter septum 1 did not go into the pore 11 10 micrometers or less, but the slurry 3 has adhered to the internal surface of pore 10 by which the aperture with which the carbon powder 2 is not filled up exceeds 10 micrometers.

[0061]

It ranks second. It is after desiccation at 110 degrees C. It calcinated at 450 degrees C and the coat layer 4 was formed. The coat layer 4 is per 11. of base materials. 150g was formed. Moreover, in this case, the carbon powder 2 with which pore 11 was filled up is burned down, as shown in drawing 14, the coat layer 4 is not formed in the pore 11 10 micrometers or less, but the aperture of the filter septum 1 inclines toward the front face of pore 10 on which an aperture exceeds 10 micrometers, and the coat layer 4 is formed.

[0062]

subsequently, the specified quantity of the dinitrodiammine platinum solution of predetermined concentration is infiltrated -- 120 degree C -- after 1-hour desiccation It calcinated at 500 degrees C for 1 hour, Pt was supported in the coat layer, and the catalyst bed was formed. The amount of support of Pt is 2g per 11. of base materials.

[0063]

With the catalyst for emission gas purification of this example obtained as mentioned above, as shown in drawing 14, an aperture inclines toward the larger pore 10 than 10 micrometers, the coat layer 4 is formed, the coat layer 4 is not formed in the pore 11 10 micrometers or less for an aperture, but lock out is prevented. Therefore, exhaust gas can pass pore 10 and pore 11, and initial pressure loss is low. Moreover, even if PM accumulates on pore 11, by the pore 10 with a larger aperture than 10 micrometers, opening area can be secured and increase of a pressure loss is prevented.

[0064]

And even if it makes [ many ] the amount of coats, since the support consistency of Pt becomes low because an aperture can control increase of initial pressure loss since the pore 11 10 micrometers or less is not blockaded, and it thickens a coat layer, the grain growth at the time of elevated-temperature durability can be controlled, and endurance improves.

[0065]

(Example 2)

It replaced with carbon powder and the catalyst for emission gas purification was prepared like the example 1 except having used the acrylic particle with a mean particle diameter of 10 micrometers.

[0066]

(Example 3)

The same DPF as an example 1 was prepared, and hexadecyl benzenesulfonic acid sodium was infiltrated. Although hexadecyl benzenesulfonic acid sodium sinks into all the pores of a filter septum, an aperture tends to flow out of big pore, and a restoration condition is maintained with surface tension by pore with a small aperture.

[0067]

Then, since the hexadecyl benzenesulfonic acid sodium with which formed the coat layer like the example 1 and it filled up in pore was burned down on that occasion, a coat layer was not formed in pore with a small aperture, but the aperture inclined toward the front face of big pore, and the coat layer was formed. And Pt was supported like the example 1.

[0068]

(Example 4)

The same DPF as an example 1 is prepared, the gas oil solution of an octylic acid cerium is infiltrated, and they are 2KPa(s) from the outflow side edge side of after that and a base material. Negative pressure

performed air suction for 2 minutes. Although the gas oil solution of an octylic acid cerium sinks into all the pores of a filter septum, an aperture tends to flow out of big pore by air suction, and a restoration condition is maintained with surface tension by pore with a small aperture.

[0069]

Then, since the gas oil solution of the octylic acid cerium with which formed the coat layer like the example 1 and it filled up in pore was burned down on that occasion, a coat layer was not formed in pore with a small aperture, but the aperture inclined toward the front face of big pore, and the coat layer was formed. And Pt was supported like the example 1.

[0070]

(Example 1 of a comparison)

The same DPF as an example 1 was prepared, the coat layer was formed like the example 1, and Pt was supported similarly. In this example of a comparison, since almost all pores were filled up with the slurry, there is a possibility that pore 10 micrometers or less may be blockaded for the aperture.

[0071]

(Example 2 of a comparison)

It replaced with the carbon powder whose mean diameter is 10 micrometers, and the catalyst for emission gas purification was prepared like the example 1 except having used the carbon powder whose mean diameter is 50 micrometers. In this example of a comparison, a catalyst bed is hardly formed in pore 50 micrometers or less for an aperture.

[0072]

(Example 3 of a comparison)

The catalyst for emission gas purification was prepared like the example 4 except having not performed air suction. In this example of a comparison, since air suction was not performed, it fills up with the gas oil solution of an octylic acid cerium in almost all pores, and a catalyst bed is almost formed in [ no ] pores. Therefore, a catalyst bed is considered that most was formed in the front face of a filter septum.

[0073]

(Example 4 of a comparison)

The same DPF as an example 1 was prepared, the polyvinyl alcohol water solution (20 % of the weight of concentration) was infiltrated, and it rinsed after that. And the coat layer was formed like the example 1 and Pt was supported. A polyvinyl alcohol water solution is filled up with this example of a comparison into all the pores of a filter septum, and a catalyst bed is almost formed in [ no ] pores in it. Therefore, a catalyst bed is considered that most was formed in the front face of a filter septum.

[0074]

<A trial and evaluation>

The floor area ratio of the pore of a filter septum was measured by the mercury porosimeter only about DPF used in the catalyst for emission gas purification of an example 1 and the example 1 of a comparison, and the example 1. A result is shown in drawing 15. With the catalyst of an example 1, the pore volume of the small pore of an aperture is large like DPF so that drawing 15 may show. This means that the small pore of an aperture was not blockaded by the catalyst bed.

[0075]

Internal pore distribution of the filter septum of each catalyst for emission gas purification and the diameter of surface average pore opening (pitch diameter of pore opening) are measured, respectively, and a result is shown in Table 1. In addition, pore distribution was measured by the mercury porosimeter and the diameter of surface average pore opening was measured by carrying out the image processing of the SEM (scanning electron microscope) photograph.

[0076]

Moreover, each catalyst for emission gas purification was packed in the case, and was catalytic-converterized. These were attached in the exhaust air system of 2L direct injection diesel power plant on the engine bench, respectively, and the pressure loss and PM elimination factor of 3 hours after when operating by 2000rpm x30Nm were measured. A result is shown in Table 1. In addition, PM elimination factor measured the weight increment of the catalyst for emission gas purification, set the remainder which subtracted it from the total amount of PM discharged from the engine to removed PM, and computed the rate to the total amount of PM.

[0077]

[Table 1]

		20-40 $\mu\text{m}$ 細孔割合 (%)	表明平均細孔開口 徑 ( $\mu\text{m}$ )	圧 損 (kPa)	PM除去率 (%)
実 施 例	1	42.3	31.2	3	85
	2	44.5	29.5	2	89
	3	47.2	24.6	4	98
	4	45	27.4	3	90
比 較 例	1	26.5	48	10	60
	2	15	58	12	70
	3	38	6	50	80
	4	40	5.8	45	83

[0078]

From Table 1, with the catalyst of the examples 1 and 2 of a comparison, the pore rate of 20-40 micrometers is low, and since many of optimal pores for the uptake of PM are blockaded by the catalyst bed, PM elimination factor is low. Moreover, although the diameter of surface average pore opening is larger than an example, a pressure loss is larger than an example. This originates in the pore rate of 20-40 micrometers being low, i.e., pore with a small aperture being blockaded by the catalyst bed.

[0079]

Moreover, with the catalyst of the examples 3 and 4 of a comparison, since the pore rate of 20-40 micrometers is comparatively high, PM elimination factor is high compared with the examples 1 and 2 of a comparison. However, the diameter of surface average pore opening has become [ the pressure loss ] very large very small therefore. Since almost all pores are blockaded with combustible material at the time of the coat stratification, this is considered to have been formed so that a coat layer might cover pore.

[0080]

However, with the catalyst for emission gas purification of each example, high PM elimination factor and a low pressure loss are compatible, and this has the high rate of 20-40-micrometer internal pore, and it is clear to originate in the diameter of surface average pore opening being fully large. 35% or more of the rate of 20-40-micrometer internal pore is desirable, and is desirable. [ especially 40% or more of ] Moreover, the diameter of surface average pore opening has desirable 10-60 micrometers, and especially its 20-40 micrometers are desirable.

[0081]

[Effect of the Invention]

That is, according to the emission-gas-purification filter of this invention, while being able to make a pressure loss low, PM collection efficiency can be made high, and a rebellion event can be reconciled. And according to the catalyst for emission gas purification of this invention which formed the catalyst bed in this emission-gas-purification filter, it is efficient, sequential combustion purification of the PM by which uptake is carried out can be carried out, and there is also no increase of a pressure loss.

[0082]

Moreover, according to another catalyst for emission gas purification of this invention, even if it forms sufficient quantity of a catalyst bed, increase of a pressure loss is controlled. Therefore, the support consistency of a catalyst metal can be made low and endurance improves by control of grain growth.

[0083]

And according to the manufacture approach of this invention, it inclines toward larger large pore than a predetermined value, and a catalyst bed can be formed certainly, easily, it is stabilized and the catalyst for emission gas purification of this invention can be manufactured.

[Brief Description of the Drawings]

[Drawing 1] It is the graph which shows the rate of a surface hole 40 micrometers or less, and the relation of a pressure loss.

[Drawing 2] It is the graph which shows the rate of the surface hole of 40-micrometer \*\*, and the relation of a pressure loss.

[Drawing 3] It is the graph which shows the rate of a surface hole 40 micrometers or less, and the relation of

PM collection efficiency.

[Drawing 4] It is the graph which shows the rate of the surface hole of 40-micrometer \*\*, and the relation of PM collection efficiency.

[Drawing 5] It is the graph which shows the rate of 20-40-micrometer internal pore, and the relation of a pressure loss.

[Drawing 6] It is the graph which shows the rate of less than 20-micrometer internal pore, and the relation of a pressure loss.

[Drawing 7] It is the graph which shows the rate of the internal pore of 40-micrometer \*\*, and the relation of a pressure loss.

[Drawing 8] It is the graph which shows the rate of 20-40-micrometer internal pore, and the relation of PM collection efficiency.

[Drawing 9] It is the graph which shows the rate of less than 20-micrometer internal pore, and the relation of PM collection efficiency.

[Drawing 10] It is the graph which shows the rate of the internal pore of 40-micrometer \*\*, and the relation of PM collection efficiency.

[Drawing 11] Surface hole distribution and internal pore distribution are the graphs which show the contribution which influences a pressure loss and PM collection efficiency.

[Drawing 12] The packer in one example of this invention is the typical sectional view of a next filter septum.

[Drawing 13] It is the typical sectional view of the filter septum after slurry sinking in in one example of this invention.

[Drawing 14] It is the typical sectional view of the filter septum after the destruction-by-fire process in one example of this invention.

[Drawing 15] It is the graph which shows the relation between an aperture and the floor area ratio of pore.

[Description of Notations]

1: Filter septum 2: Carbon powder 3: Slurry 4: Coat layer

10: Pore to which an aperture exceeds 10 micrometers 11: An aperture is pore 10 micrometers or less.

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[Translation done.]

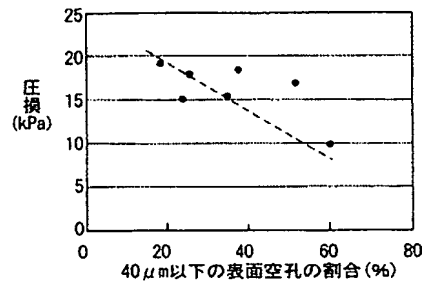
## \* NOTICES \*

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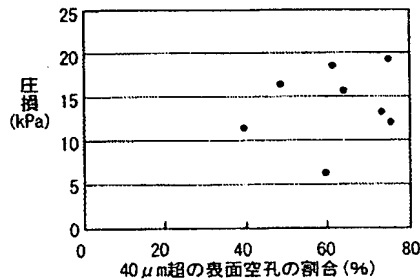
1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. \*\*\*\* shows the word which can not be translated.
3. In the drawings, any words are not translated.

## DRAWINGS

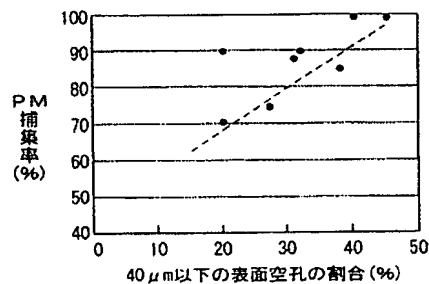
[Drawing 1]



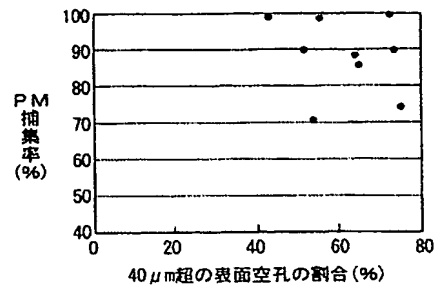
[Drawing 2]



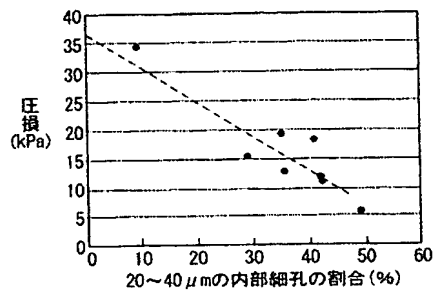
[Drawing 3]



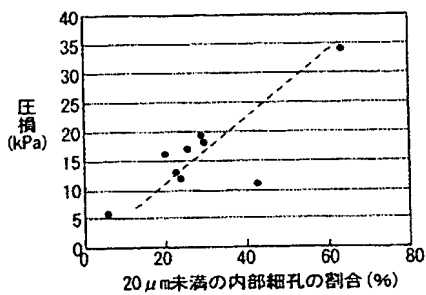
[Drawing 4]



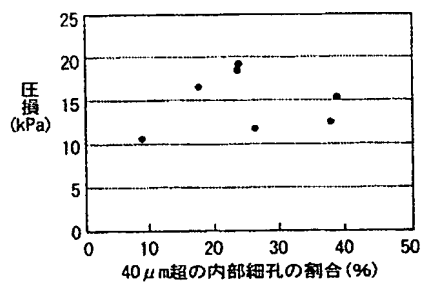
[Drawing 5]



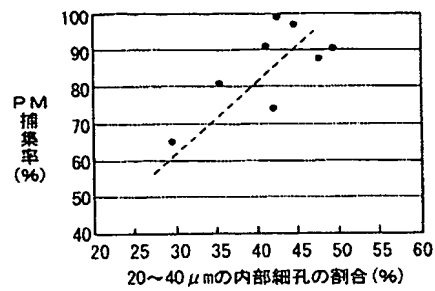
[Drawing 6]



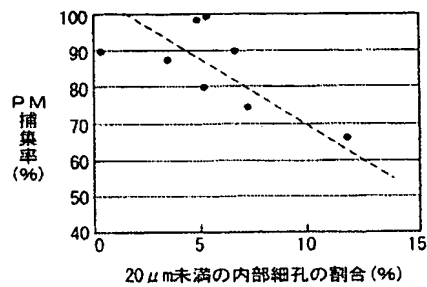
[Drawing 7]



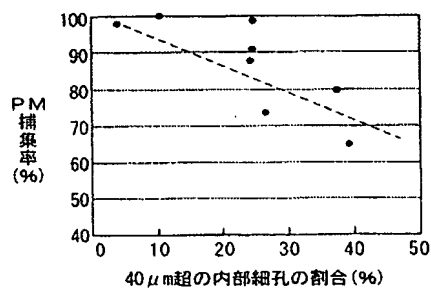
[Drawing 8]



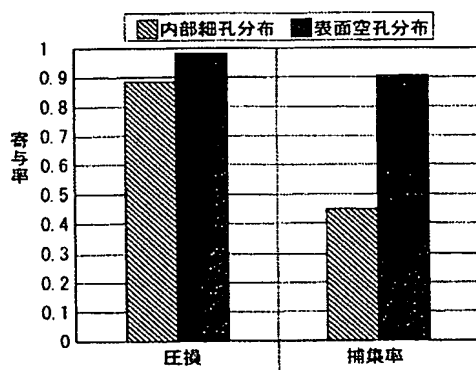
[Drawing 9]



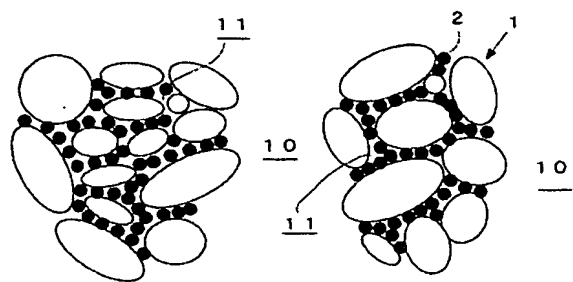
[Drawing 10]



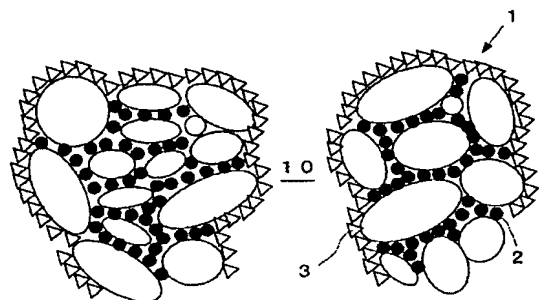
[Drawing 11]



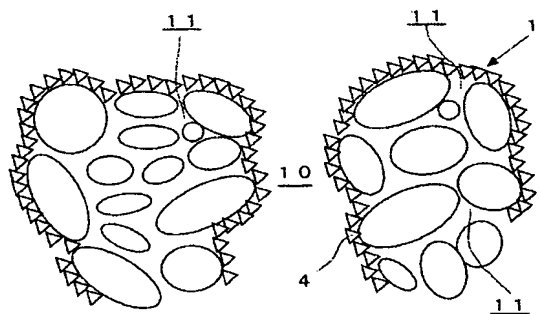
[Drawing 12]



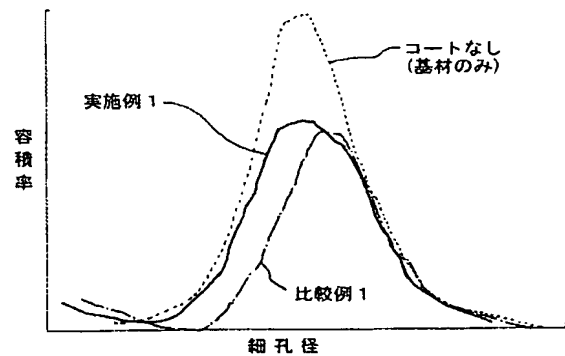
[Drawing 13]



[Drawing 14]



[Drawing 15]



[Translation done.]